Volume expansion resulting from localized melting or freezing of the confined zone is accommodated by the volume expansion space or slot. After at least one traverse, this slot will partially fill and form a ridge on the finished ingot. This ridge is easily removed by mechanical processing such as grinding and polishing.

Normally solid (i.e., solid under standard temperature and pressure conditions) material having a substantially round cross section and which evidences a volume change upon phase change is zone refined to substantially remove impurities therefrom by a process which comprises:

(a) containing a charge or ingot of the material in a cylindrical cavity **11** defined by a container **10** having a volume expansion space **12** along the length of the container;

(b) traversing from one end of the ingot to another at least once a zone of a predetermined elevated temperature sufficient to melt a portion of the material, thereby sweeping impurities to one end of the ingot; and

(c) accomodating expanded material in the volume expansion space **12**, whereby pressure buildup is eliminated, fractures and other damage to the zone refined material or the container **10** are minimized and the substantially round cross section of the ingot is retained.

The material of the container **10** may comprise any refractory material such as borosilicate glass, silica, alumina and the like. The length and inside diameter dimensions of the container are chosen to just contain a substantially cylindrical ingot which is to be zone refined.

The width of the slot **12** is related to the size of the ingot. The minimum width is that which may be obtained by any convenient process such as wire saw and the like. The maximum width of the slot is dictated by conditions of the final ingot after zone refining. It is desired that the ingot retain its substantially full round cross section after zone refining. The larger the ingot, the wider the slot may be. As an example, for zone refining an ingot of thallium bromoiodide having a diameter of 6 mm, a slot width of 1 mm is adequate to retain the substantially round cross section of the fiber.

The thickness of the container is immaterial other than it be thick enough to withstand normal handling and not so thick that it adversely affects thermal gradients. As is well-known, steep thermal gradients are desired for zone refining. For zone refining thallium bromoiodide, which melts at about 414° C., a four-turn heating coil 1 cm wide generates a thermal gradient of about 100°/mm, which is considered adequate. For a 6 mm diameter ingot, a wall thickness of the container of about 0.5 to 0.7 mm is sufficient to meet the foregoing criteria.

Plugs **15,16** may comprise the same material as the container or the same material as the ingot being zone refined so long as, in the latter case, the plugs are not passed through the heating coil.

A cast ingot of substantially round cross section is used to charge the container. Powdered material has been found to result in a finished product having too many voids. However, loose power may be sprinkled in the volume expansion space in order to compensate for voids between the ingot and the container and to ensure a substantially fully round cross section of the finished ingot.

In order to compensate for matter transport caused by volume change during phase change and by surface tension, the container is inclined at a slight angle determined by the properties of the material; see, e.g., the Pfann reference, pp. 48–50.

## EXAMPLE

A mixed melt ingot of thallium bromoiodide, prepared from a mixture of 45.7 mole percent TlBr and 54.3 mole percent TlI and in the form of a cylinder 100 mm long and 6 mm diameter, was zone refined in apparatus similar to that depicted in the FIGURE. A quartz container of length 140 mm and ID 6.1 mm was employed, with a slot of 0.6 to 1.0 mm in width running the length of the container. End plugs of thallium bromoiodide were employed. A small amount of thallium bromoiodide powder was sprinkled substantially uniformly along the slot to ensure substantially fully round cross section of the finished crystal.

A four-turn heating coil 1 cm wide traversed twenty-three times along the length of the container. A thermal gradient of about 100° C./mm and a temperature of about 500° C. in the zone were established. It was found that the combined effects of phase change and surface tension caused a forward matter transport. To compensate, the container was tilted at about 6°, making the climb at this small angle during its slow forward motion.

Upon completion of the zone refining operation, the crystal was removed from its container. A ridge of expanded material, running along the length of the crystal, was easily removed by snipping off.

Prior to zone refining, the crystal was of cloudy appearance; following twenty-three passes of the molten zone, a crystal of exceptional clarity was obtained. As is well-known, the transparency of thallium bromoiodide crystals is adversely affected by the presence of impurities.

Thus, an alkali halide crystal of substantially round cross section and which expands upon melting was purified without damage to the crystal or the container. The substantially round cross section of the crystal was retained.

What is claimed is:

**1**. A zone refining process for substantially removing impurities from a mixed thallium halide material which comprises:

(a) containing a charge of the material in a cylindrical cavity defined by a container maintained substantially horizontal and having a volume expansion space along the length of said container and above said cavity, said volume expansion space having a width no wider than that necessary to retain a substantially round cross-section of said charge following zone refining;

(b) traversing from one end of said charge to another at least once a zone of a predetermined elevated temperature sufficient to melt a portion of said material, thereby sweeping impurities to one end of said charge; and

(c) accomodating expanded material in said volume expansion space, whereby pressure buildup is eliminated, fractures and other damage to the zone refined material or its container are minimized and said substantially round cross-section is retained.

**2**. The process of claim **1** in which following said zone refining process, the expanded portion of said material is removed from said charge.

**3**. The process of claim **1** in which said crystal consists essentially of TlBr and TlI, in proportions ranging from about 40 to 45 mole percent TlBr, balance TlI.

* * * * *